

DATA VALIDATION AND USABILITY REPORT

Polycyclic Aromatic Hydrocarbons (PAHs) by USEPA SW-846 Method 8270D-SIM

Total Organic Carbon (TOC) by Plumb 1981 (Combustion Infrared) for Sediments

Pentachlorophenol (PCP) by USEPA SW-846 Method 8041A

Mercury by USEPA SW-846 Methods 7470B/7471A

Total Solids by Method SM2540G

Grain Size by Method PSEP

Project: East Eagle Harbor OU 2016 OMMP Monitoring
Sediments January 2017

Project/Task Number: 10049950-3

Sample Data Package: 17A0304

Laboratory: Analytical Resources, Incorporated (ARI)

Sample Matrix: Sediment, Water

Sampling Dates: 23-24 January 2017

Validation Guidelines: Project QAPP (*Final 2016 Quality Assurance Project Plan Analytical Quality Assurance Plan, East Harbor Operable Unit, Wyckoff/Eagle Harbor Superfund Site* [January 9, 2017]), United States Environmental Protection Agency (USEPA) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd edition (SW-846); USEPA *National Functional Guidelines for Superfund Organic Methods Data Review*, OLEM 9355.0-134, EPA-540-R-2016-002 [September 2016]), USEPA *National Functional Guidelines for Inorganic Superfund Methods Data Review*, OLEM 9355.0-133, EPA-540-R-2016-001 [September 2016]), USEPA-540-R-10-011, January 2010; USEPA *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use*, USEPA 540-R-08-005, January 13, 2009; and professional judgment

Validation Level: Stage 2BVM (100%), Stage 4VM (10%)

Data Reviewer: Lynn K. Lutz, HDR

Sample ID	Matrix	Lab ID	PAHs	TOC	PCP	Mercury	Total Solids	Grain Size
012317001	Sediment	17A0304-01	X	X	X	X	X	X
012317002	Sediment	17A0304-02	X	X	X	X	X	X
012317003	Sediment	17A0304-03	X	X	X	X	X	X
012317004	Sediment	17A0304-04	X	X	X	X	X	X
012417001	Sediment	17A0304-05	X	X	X	X	X	X
012417002	Sediment	17A0304-06	X	X	X	X	X	X
012417003	Sediment	17A0304-07	X	X	X	X	X	X
012417004	Sediment	17A0304-08	X	X	X	X	X	X
012417004 MS	Sediment	17A0304-08	X					
012417004 MSD	Sediment	17A0304-08	X					
012317005W	Water	17A0304-09	X			X		

SUMMARY

All laboratory data were acceptable with qualification.

I. SAMPLE RECEIPT / CHAIN OF CUSTODY

The chains of custody (COCs) were filled out and signed. Samples were received within the correct temperature range of 0-6°C. No qualification was required.

II. HOLDING TIMES

Holding time criteria were met. No qualification was required.

PAHs: Samples were extracted within 14 days of collection (seven days for the water sample) and analyzed within 40 days of extraction. No qualification was required.

TOC: Samples were analyzed within 14 days of collection. No qualification was required.

PCP: Samples were analyzed within 14 days of collection. No qualification was required.

Mercury: Samples were analyzed within 28 days of collection. No qualification was required.

Total Solids: Samples were analyzed within 14 days of collection. No qualification was required.

Grain Size: Samples were analyzed within 2 months of collection. No qualification was required.

III. INSTRUMENT CALIBRATION

INITIAL CALIBRATIONS

PAHs: Initial calibration criteria were met. The relative standard deviation (RSD) of the curve was $\leq 20\%$. The low standard concentration was at the RL. No qualification was required.

TOC: Initial calibration information was not included in the data package, as it is stored in the instrument. No qualification was required.

PCP: Initial calibration criteria were met. The RSD of the curve was $\leq 20\%$ and the low standard was below the RL. No qualification was required.

Mercury: Initial calibration criteria were met. The correlation coefficient of the curve was ≥ 0.990 and the low standard was below the RL. No qualification was required.

Total Solids: Analysis is gravimetric; calibration is not applicable.

Grain Size: Analysis is gravimetric; calibration is not applicable.

SECOND SOURCE STANDARDS

PAHs: Second source standard criteria were met. All analytes had %D values less than 20%. Some of the ICV forms had incorrect true values listed for the total benzofluoranthenes and the %D values appeared to be outside control limits, but compared to the corrected true values, the results were within control limits. No qualification was required.

TOC: Second source standard criteria were met. The %D values were less than 10%. No qualification was required.

PCP: Second source standard criteria were met. All %D values were less than 20%. No qualification was required.

Mercury: Second source standard criteria were met. All %D values were less than 20%. No qualification was required.

Total Solids: Analysis is gravimetric; calibration is not applicable.

Grain Size: Analysis is gravimetric; calibration is not applicable.

CONTINUING CALIBRATIONS

PAHs: Continuing calibration criteria were met. All analytes had %D values less than 20%. No qualification was required.

TOC: Continuing calibration criteria were met. The %D values were less than 10%. No qualification was required.

PCP: Continuing calibration criteria were met. All %D values were less than 20%. No qualification was required.

Mercury: Continuing calibration criteria were met. All %D values were less than 20%. No qualification was required.

Total Solids: Analysis is gravimetric; calibration is not applicable.

Grain Size: Analysis is gravimetric; calibration is not applicable.

IV. BLANKS

Target analytes were not detected in the blanks except as noted below.

METHOD BLANKS (MB)

PAHs: Target PAHs were not detected in the MBs. No qualification was required.

TOC: TOC was detected below the MRL. Sample results were greater than 10 times the blank and no qualification was required.

PCP: PCP was not detected in the MBs. No qualification was required.

Mercury: Mercury was not detected in the MBs. No qualification was required.

Total Solids: The MB was non-detect. No qualification was required.

Grain Size: Not applicable.

INITIAL AND CONTINUING CALIBRATION BLANKS (ICB and CCB)

PAHs: Not applicable.

TOC: TOC was detected below the MRL. Sample results were greater than five times the blank and no qualification was required.

PCP: Not applicable.

Mercury: Mercury was detected at trace levels in the ICBs and CCBs. Sample results were greater than five times the blank and no qualification was required.

Total Solids: Not applicable.

Grain Size: Not applicable.

EQUIPMENT BLANKS (EB)

Sample 012317005W was collected as an EB.

PAHs: Acenaphthene was detected at a trace level in the EB. Sediment sample results were more than five times the blank concentration, and the blank results is not expected to have impacted the sample results. No qualification was required.

TOC: The EB was not analyzed for TOC. No qualification was required.

PCP: The EB was not analyzed for PCP. No qualification was required.

Mercury: Mercury was not detected in the EB. No qualification was required.

Total Solids: The EB was not analyzed for total solids. No qualification was required.

Grain Size: Not applicable.

V. LABORATORY CONTROL SAMPLES (LCS) and LABORATORY CONTROL SAMPLE DUPLICATES (LCSD), and STANDARD REFERENCE MATERIAL (SRM)

LCS/LCSD

PAHs: LCS recoveries were within the project control limits. No qualification was required.

TOC: LCS analysis was not performed; an SRM was analyzed instead.

PCP: LCS recoveries were within the control limits. No qualification was required.

Mercury: LCS recoveries were within the control limits. No qualification was required.

Total Solids: Not applicable.

Grain Size: Not applicable.

SRM

PAHs: A number of SRM recoveries were below acceptable limits, and the results for these analytes in the sediments have been qualified as estimated (J) or non-detect estimated (UJ) as show in the table below.

Analyte	SRM Recovery	SRM Recovery Limits	Qualifier
Fluorene	12.7%	44.1-155%	J / UJ
Phenanthrene	64.1%	85.1-115%	J / UJ
Anthracene	40.9%	60.5-140%	J / UJ
Pyrene	39.8%	50.2-150%	J / UJ
Benzo(a)anthracene	33.3%	48.2-152%	J / UJ

TOC: Recovery of the SRM was within control limits. No qualification was required.

PCP: An SRM was not analyzed. No qualification was required.

Mercury: Recovery of the SRM was within control limits. No qualification was required.

Total Solids: Not applicable.

Grain Size: Not applicable.

VI. MATRIX SPIKE / MATRIX SPIKE DUPLICATE (MS/MSD)

Sample 012417004 was designated on the COC for MS/MSD analysis for PAHs only.

PAHs: A number of MS and/or MSD recoveries and/or RPDs were outside control limits, and the results for these analytes in the parent sample have been qualified as estimated (J) or non-detect estimated (UJ) as show in the table below.

Analyte	MS/MSD Recoveries	MS/MSD Recovery Limits	RPD	RPD Limit	Qualifier
Naphthalene	20.5%, OK	36-120%	88.1%	30%	J
2-Methylnaphthalene	32.7%, OK	35-120%	33.2%	30%	J
1-Methylnaphthalene	34.9%, OK	39-120%	40.9%	30%	J
Phenanthrene	39.9%, OK	46-120%	OK	NA	J
Anthracene	-1.11%, 19.0%	36-120%	OK	NA	J
Fluoranthene	39.7%, 40.8%	46-120%	OK	NA	J
Pyrene	0.1%, OK	49-120%	OK	NA	J
Chrysene	20.6%, 16.3%	48-120%	OK	NA	J
Total Benzofluoranthenes	39.6%, OK	46-120%	OK	NA	J

TOC: The lab spiked sample 012417004. Only an MS was spiked, and the recovery was within control limits. No qualification was required.

PCP: The lab spiked sample 012417004. Recoveries and the RPD were within the control limits. No qualification was required.

Mercury: The lab spiked sample 012417004. Recoveries and the RPD were within the control limits. No qualification was required.

Total Solids: Not applicable.

Grain Size: Not applicable.

VII. DUPLICATES/REPLICATES

FIELD REPLICATES

Field replicate samples were collected and identified in the following table.

Field Duplicate Sample	Parent Sample	Matrix	Analyses
012417003	012417002	Sediment	PAHs, TOC, PCP, Mercury, Total Solids, Grain Size

Qualifiers were assigned to duplicate and parent samples for the specific analytes that fail the %RPDs. If the analyte was undetected then the qualifier assigned was UJ.

PAHs: Three results were outside the 30% control limit and required qualification, as shown in the table below. The qualified results for benzo(a)anthracene are both well below above the Intertidal Sediment Carcinogen remedial goal and well below the other remedial goals and standards listed in the QAPP and are fully usable. The other qualified results are well below the remedial goals and standards listed in the QAPP and are fully usable.

Analyte	Parent Result	FD Result	RPD	Qualifier
Benzo(a)anthracene	712 µg/kg	972 µg/kg	31%	J / J
Fluoranthene	64.4 µg/kg	69.1 µg/kg	49%	J / J
Anthracene	279 µg/kg	777 µg/kg	94%	J / J

TOC: FD results were outside the 20% control limit and required qualification, as shown in the table below. There are no remedial goals and standards listed in the QAPP, and the qualified results are fully usable.

Analyte	Parent Result	FD Result	RPD	Qualifier
TOC	1.52%	1.98%	27%	J / J

PCP: The parent and FD results for PCP were both non-detect. No qualification was required.

Mercury: Field duplicate results were within the 20% control limit. No qualification was required.

Total Solids: The FD result at 2% RPD was within the control limit of 20%. No qualification was required.

Grain Size: Field duplicate results were within the 20% control limit. No qualification was required.

LABORATORY DUPLICATES

PAHs: Not applicable.

TOC: Lab triplicate analysis was performed sample 12417004. RPDs between each parent/replicate result and the RSD for all three results were below the control limit of 20%. No qualification was required.

PCP: Lab duplicate analysis was not performed on any samples from this project. No qualification was required.

Mercury: Lab duplicate analysis was performed on the EB (sample 012317005W) and sediment sample 012417004. The result for the EB were within control limits. The RPD for the sediment was

outside the 20% control limit and required qualification, as shown in the table below. Both qualified results are below the sediment standards listed in the QAPP and are fully usable.

Analyte	Parent Result	Lab Duplicate Result	RPD	Qualifier
Mercury (012317005W)	0.131 mg/kg	0.1048 mg/kg	22%	J / J

Total Solids: Lab triplicate analysis was performed sample 12417004. RPDs between each parent/replicate result and the RSD for all three results were 4% or less, within the control limit of 20%. No qualification was required.

Grain Size: Lab triplicate analysis was performed sample 12417004. RPDs between each parent/replicate result and the RSD for all three results were 5% or less, within the control limit of 20%. No qualification was required.

VIII. INTERNAL STANDARDS (ISTD) AND SURROGATES

All ISTD and surrogate criteria were met except as noted below.

PAHs: ISTD areas and retention times and surrogate recoveries were within control limits. No qualification was required.

TOC: Not applicable.

PCP: All surrogate recoveries were within the control limits. No qualification was required.

Mercury: Not applicable.

Total Solids: Not applicable.

Grain Size: Not applicable.

IX. POST DIGESTION SPIKE AND DILUTION TEST

Post-digestion spike (PDS) analyses and serial dilution tests were performed for Method 6010C and 6020A metals. PDS results were within the control limits of 75-125% except as noted below. The dilution tests were not applicable unless sample results were greater than 50 times the MDL.

Mercury: Not applicable.

X. INTERFERENCE CHECK SAMPLES (ICS)

ICS results were applicable to the Method 6010C and 6020A metals analyses. Results were within control limits of 80-120% except as noted below.

Mercury: Not applicable.

XI. REPORTING LIMITS (RL) AND METHOD DETECTION LIMITS (MDL)

The MDLs were \leq one-half the RLs. RLs should be below the remedial goals and standards listed in the QAPP.

PAHs: The RLs were below the remedial goals and standards listed in the QAPP. The MDLs were \leq one-half the RLs. No qualification was required. A number of results were reported from diluted

analyses due to high concentrations. The RLs for the diluted analyses were also below the remedial goals and standards, and no non-detect results were reported from diluted analyses.

TOC: The RL of 0.02% matches the required RL listed in the QAPP.

PCP: The nominal RL of 6.25 µg/kg is well below the remedial goals and standards, and no non-detect results were reported from diluted analyses.

Mercury: The RLs were below the remedial standards listed in the QAPP. The MDLs were ≤ one-half the RLs. No qualification was required.

Total Solids: The RL was below the required RL listed in the QAPP. There were no remedial goals and standards listed for this analysis.

Grain Size: The RL was below the required RL listed in the QAPP. There were no remedial goals and standards listed for this analysis.

XII. SAMPLE RESULTS / TRANSCRIPTION VERIFICATION

Transcription between the data package and the EDDs was verified. Sample results reported between the MDL and RL were qualified as estimated (J).

PAHs: No issues.

TOC: No issues.

PCP: The RPD between the primary and conformation results in sample 12317003 at 75% was above the requirement of 40%. The result for this sample has been qualified as estimated (J). The qualified result was well below the remedial goals and standards and is fully usable.

Mercury: No issues.

Total Solids: The data package contained results for total solids by method PSEP 1986 as well as by SM2540C. The results for SM2540C only were reported in the EQUIS file.

Grain Size: The results were reported as percent retained in each size fraction. The total percentages for each sample did not add up to exactly 100%. For samples in which the total percentage was not within 5% of 100%, the results have been qualified as estimated (J) as shown in the table below.

Sample ID	Total percent	Qualifier	Sample ID	Total percent	Qualifier
012317001	106.7%	J	012417002	122.5%	J
012317002	112.8%	J	012417003	123.1%	J
012317003	111.8%	J	012417004	122.6%	J
012317004	110.6%	J	012417004 DUP1	123.1%	J
012417001	106.7%	J	012417004 DUP2	120.9%	J

XIII. PARCCS PARAMETERS AND DATA USABILITY

Data were usable as discussed below.

The data quality indicators (DQIs), expressed in terms of precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS parameters), were assessed. This included the evaluation of sample integrity, holding times, trip blanks, rinsate

blanks, method blanks, internal standards, surrogate recoveries, MS/MSD recoveries, LCSs, and field duplicate precision. The results of the DQI assessment are provided below.

PRECISION

Field replicates are collected to assess sampling precision. Lab duplicates, LCSDs, and MSDs are analyzed to assess analysis precision. Precision is best expressed in terms of relative percent difference (RPD).

The precision goals were generally met. When RPDs were above the control limits, this may be an indication of sample non-homogeneity. The qualified results were well below the remedial goals and standards and are fully usable.

ACCURACY

Accuracy or bias is measured through the analyses of LCSs, MSs, MSDs, and SRMs. Sample specific accuracy is measured through surrogate recovery. Accuracy is expressed as percent recovery (%R). Bias may be indicated if analytes are detected in blanks.

Accuracy goals were generally met. LCS recoveries and surrogate recoveries were within control limits. Blanks were generally non-detect and did not introduce bias.

Some SRM recoveries were below control limits, suggesting that sample results may be biased proportionately low.

REPRESENTATIVENESS

Representativeness refers to the degree sample data accurately and precisely describes the population of samples at a sampling point or under certain environmental conditions. Samples that are not properly preserved or are analyzed beyond holding times may not be considered representative. Review of sampling procedures, laboratory preparation, analysis holding times, trip blank and rinsate blank analysis help in providing this assessment.

Representativeness goals were met. Samples were collected at predetermined locations according to the project plan. Some blanks had only trace levels of contaminants detected that did not bias the sample results.

COMPARABILITY

The selection of standardized methods and consistent laboratory practices facilitates the comparison of data between sampling events.

Comparability goals were met. Samples were analyzed by standard methods as in past events. Results were reported down to the RLs.

COMPLETENESS

Completeness is determined for both field and analytical objectives. Field completeness is calculated from the number of samples proposed versus the actual number of samples collected. Analytical completeness is expressed in terms of usable data.

Completeness goals were met. No data were rejected, and completeness for all analytes was 100%.

SENSITIVITY

Analytical sensitivity is the concentration at which the measurement system can quantitate target analytes in the environmental matrices of concern. Analytical sensitivity is expressed in terms of the RL, which is provided by the respective laboratories as their reasonable and

defensible quantitation limit for environmental samples above the MDL, which is established by each laboratory using pure water or clean matrix.

Sensitivity goals were met. The MDLs were \leq one-half the RLs. RLs were at or below the remedial goals and standards listed in the QAPP.

PAHs: No data were rejected. Data required minimal qualification. All data are usable as qualified.

TOC: No data were rejected. Data required minimal qualification. All data are usable as qualified.

PCP: No data were rejected. Data required minimal qualification. All data are usable as qualified.

Mercury: No data were rejected. Data required minimal qualification. All data are usable as qualified.

Total Solids: No data were rejected. Data required minimal qualification. All data are usable as qualified.

Grain Size: No data were rejected. Data required minimal qualification. All data are usable as qualified.